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Reflection Spectra of Bio-organic Materials in the 2.5-4 μ Region and the Interpretation of the Infrared Spectrum of Mars

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I. Introduction

The nature of the Martian surface and the possibility of life existing there has for decades stimulated considerable speculation and research. A notable contribution to the rather meager quantitative data on the planet was made recently by Sinton (1,2,3) when he observed "absorption bands" in the 3-4 micron radiation reflected by the dark areas, and which were less pronounced in the radiation from the light areas. The spectral curves he obtained using the 200 in. telescope at Mt. Palomar Observatory are shown in Fig. 1. Due to the low available energy the resolution on the disc was poor, being at best half a planetary diameter, and the accepted area for the nominal Syrtis Major spectrum in fact included large expanses of bright areas. The associated spectral resolution calculated from the slit width and dispersion was 90 cm^{-1} (0.11 μ) for some of the recordings and 45 cm^{-1} (0.056 μ) for the remainder.

The significant observation was of a structure, more pronounced for the dark areas than for the bright ones. Sinton considered the structure to be absorption bands and due to surface components. The bands, located at 2710 (3.69 μ), 2793 (3.58 μ) and 2910 cm⁻¹ (3.45 μ), fall in the region where organic molecules, and some inorganic compounds, absorb. Inorganic carbonates were eliminated because of a lack of spectral correspondence, but the possibility that some other inorganic compound may be responsible was not discarded. However, Sinton considered the spectra to resemble closely those of organic molecules and of plants. The presence of carbohydrates was inferred from the 2710 cm⁻¹ band, which he stated is present for this class of compounds and for the alga Cladophora. While not totally rejecting an inorganic interpretation Sinton evidently believes this is a remote possibility and that "the presence of large organic molecules is indicated...."

A variant to this interpretation has been advanced by Colthup (4) who noted that the 2710 cm⁻¹ band is unique for a C-H group in having such a low wavenumber. Not only is this band rare in plants, as Sinton commented,

it is also very uncommon for organic compounds in general. The only structural component having an intense band at this wavenumber is the CH of an aldehyde group. From the high relative intensity of this band in the Martian spectra the aldehyde CH's must have a high concentration relative to other CH's. The only molecule which is suitable is acetaldehyde, which also has a second band that could be contributing to the 2793 cm⁻¹ absorption. The remaining 2793 cm⁻¹ absorption and all of that at 2910 cm⁻¹ is then due to other CH groups of the Martian organic matter. Colthup speculates that "acetaldehyde may be an end product of certain anaerobic metabolic processes." However it has been pointed out (5) that the high volatility of this chemical would ensure a high concentration in the atmosphere, so that the observed bands would have to be due to gaseous acetaldehyde. It should then be observed over the entire disk and not restricted to certain areas.

The implications of Sinton's observations and the interpretations are clearly quite great. The potential demonstrated for infrared probing of the Martian surface is such that an infrared spectrometer has been proposed for inclusion in spacecraft experimental packages destined for Mars. It was to record laboratory reflection spectra on a wide variety of terrestrial objects in order to facilitate interpretation of the Martian observations, that the present investigation was initiated. Shortly after its inception the scope of the project was broadened to include an extensive examination of the nature and interpretation of Sinton's observed spectra. The present paper is a description of part of this work.

II. Theory

No attempt will be made here to develop a comprehensive theory which would relate an observed planetary reflection spectrum to the absorption frequencies and morphology of the surface material. However, there is one point that must be stressed, a point that has not been properly appreciated heretofore.

There is a fundamental difference in appearance between absorption and reflection spectra. The reflection spectrum corresponding to a simple absorption band will have a minimum on the high wavenumber side of the resonant frequency, and a maximum which lies on the low wavenumber side for weak bands but which broadens and shifts to the other side as the intensity of the associated absorption band increases. The relation between absorption and reflection for a single strong organic band is shown in Fig. 2. The

transmission measurement was made on a 25 μ film of lucite. A thick block of the plastic, having essentially zero transmission and a smooth reflecting surface, was used to obtain the reflection spectrum.

Now the relationship discussed above, is valid only for smooth surfaces and for observations at a single reflection angle on materials which are thick enough to absorb completely (specular reflection). As the surface is roughened some of the radiation will pass through parts of the material before being reflected. The result will be a blend of absorption and reflection features, the exact proportions being a function of the surface configuration and the dielectric properties of the solid. When the observed intensity includes reflection at a variety of angles the sharpness of the relationship between the S-type reflection curve and the absorption band is further blurred. Optically thick materials will exhibit unique effects due to reflection at the second interface. These complications render the interpretation of the reflection spectrum of an unknown body, unknown both in morphology and composition, ambiguous. And of course this situation applies to the Martian spectra.

With respect to Sinton's observations it is not possible to say whether the features are predominantly absorption or reflection, since the resolution was too low to produce definitive contours. As a result an additional uncertainty is introduced which, together with the estimated probable error of 16 cm^{-1} , leads to the following ranges for the associated vibrational wavenumbers--2674-2726, 2754-2809, and 2834-2925 cm⁻¹. These will throughout the remainder of this paper be referred to as the Mars I, II, and III bands respectively.

Because of these complexities we have not attempted to calculate these interactions from first principles but rather have determined empirically the spectral characteristics of a variety of materials under various conditions.

III. Experimental

A. Apparatus

For the spectral observations we have used a Beckman IR-7 spectrophotometer. Transmission measurements were made on samples prepared in a variety of ways. Powders were observed using the standard KBr technique, algae and bacteria were deposited in thin films on a AgCl plate, lichen such as Evernia were pressed into thin, rather heterogeneous, sheets, or ground with KBr and pelleted, and

some samples such as the epidermis and cuticle of Agapanthus were examined directly. Generally no attempt was made to make the transmission measurements quantitative.

The reflection measurements used two different experimental arrangements. In one the standard Beckman specular reflection attachment was placed in the sample beam. All samples were examined in this manner with the angles of incidence and reflection both equal to 30° . To protect the samples from heating by the beam, and to keep unwanted radiation from striking the detector for the wide slits required, we interposed band-pass filters between the source and sample. Since the reflected energy of the sample was invariably very low the reference beam was attenuated by screens in order to obtain a convenient recorder reading. These screens were calibrated in the actual combinations used by employing the single-beam feature of the IR-7. Since the screens' transmissions varied with wavelength the 100 per cent lines were in effect tilted. This effect has not been factored out of the spectra presented in this paper, but the appearance of the bands is still sufficiently faithful for our present purpose.

There are no quantitative reflection coefficients given since the Beckman attachment was designed to observe specular reflection only. In the case of diffuse reflection it is important to know the solid angle which is accepted by the detecting apparatus, and this is not simply calculable for this experimental arrangement. Moreover since the slit width is not constant but varies with wavenumber the solid angle and hence the recorded intensity is also wavenumber dependent. The resulting spectra are hence only qualitative.

The second means of recording the reflection spectra was a single beam attachment, figure 3, which we constructed to enable us to observe at angles of incidence and reflection from 5° to 80° and to obtain quantitative reflection coefficients. The front surface of the sample is imaged on the entrance slit with f/10 optics so that the accepted solid angle is 7.8 x 10⁻³ steradians. Incorporated in the device was a film polarizer graciously loaned by Dr. George Bird of the Polaroid Corp. Thus we were equipped to study both the intensity and polarization of the reflected radiation. To date only a few of our samples have been examined with this accessory.

B. Sample Types

In the course of our studies a large number of varying types of samples have been examined. They included pure organic chemicals, polymers, biological specimens, pure inorganic compounds and mineralogical samples, the latter two ground to a particle size \langle 0.1 mm and then pelleted. We felt this would produce spectra more valid for comparison with the Martian spectra than polished surfaces. No effort was made to control the particle size. The effect of this factor on the spectra is undoubtedly important and merits a careful study.

IV. Results and Discussion

Of the many spectra recorded only a few, demonstrating specific points, will be presented here. Actually there was a considerable similarity within individual classes of compounds and the major groupings of the plants.

The reflection spectrum of paraffin wax, figure 4, is primarily a superposition of two S-type curves similar to that in figure 2. The energy minima of the curves are shifted about 18 cm⁻¹ toward higher wavenumbers from the positions, 2850 and 2920 cm⁻¹, of the absorption bands.

Cellulose, a major constituent of plants, has a simple reflection spectrum with a strong minimum at 2900 cm⁻¹ and a weak shoulder near 2750 cm⁻¹, fig 5. The latter has far too low an intensity relative to the 2900 cm⁻¹ absorption to account for the Mars I or II bands since this would require a much more intense Mars III band. While the reflection from the related carbohydrates - starch and sugar, show little structure, even the absorption showed only a weak band at 2750 cm⁻¹ relative to the major absorption between 2800 and 3000 cm⁻¹.

Sinton, in suggesting that the Mars I band be due to carbohydrates, has presented the reflection spectrum of the alga <u>Cladophora</u> exhibiting a minimum near 2750 cm⁻¹. Our spectrum of the same alga reveals only a very weak minimum at 2750 cm⁻¹ with much stronger minima at 2860 and 2935 cm⁻¹. The relative intensity of the Mars I band is far too high for Sinton's interpretation and, if it is to be attributed to organic matter, it must be assigned to the CH of free aldehyde groups. In carbohydrates such groups are present in trace amounts and would not be expected to produce intense spectral features.

Lichens have been a prime candidate for explaining some of the optical observations of Mars (e.g. Kuiper (7)), although Salisbury (7) has recently

demonstrated the weaknesses in this interpretation. Our spectra of lichen are all quite similar in having minima near 2875 and 2935 cm⁻¹, with perhaps a very faint one near 2715 cm⁻¹. The intensities are such that they could account for only the Mars III band, leaving the other two to be assigned to other surface components.

One of the most interesting biological specimens studied is the flat smooth leaf of the lily Agapanthus. The reflection spectra of both this leaf and that of the prickly-pear cactus show a marked similarity to that of paraffin wax. The reflection spectrum, figure 6, is characterized by two s-type curves, which correspond to the two absorption bands observed for the epidermis and cuticle in transmission, fig. 7. For these samples the surface waxy layer is smooth enough to give nearly specular reflection, together with true reflection features.

The interest of this observation is twofold. First it demonstrates that biological objects can have reflection features of the pure reflection type as well as of the absorption type. Second, <u>Agapanthus</u> and the cactus are excellent examples of species which have a protective casing which can keep a specific component, in these instances water, from passing directly into or out of the leaf. Such membranes could be an important feature of a Martian biology.

Of the inorganic samples studied only the carbonates show much promise. Generally they have strong bands in the 2800-3000 cm⁻¹ region, e.g. CaCO₃, fig. 8. It is relatively easy to assign the Mars III band, the compounds MgCO₃, FeCO₃, CaCO₃, CaMg(CO₃)₂, ZnCO₃, BaCO₃ and CdCO₃ all having bands within the estimated range. The Mars II band is more difficult to interpret with only BaCO₃, PbCO₃, and CdCO₃ as possibilities. Now, however we encounter problems of cosmic abundance (8). For the Mars III band only PbCO₃, fig. 9, comes close, with a band at 2749 cm⁻¹, but one would hardly expect a lead salt as the dominant cover over extensive areas of a planet.

The situation is then quite unsatisfactory. Assigning the Mars III band to either inorganic carbonates or organic compounds is straightforward. The other two bands remain without a satisfactory interpretation. If they are due to organic matter both must be assigned to aldehyde CH groups, and the relative concentration must be very high. No assignment based on observed spectra has been proposed that appears satisfactory. On the other hand, inorganic carbonates are improbable, due primarily to the low

cosmic abundance of certain necessary elements such as lead.

V. Significance of Surface Emission

In his first paper Sinton (1) discussed in considerable detail the possible contributions of Martian thermal emission to the observed intensities. For the 1956 observations, for which there was no spatial resolution, he estimated an effective temperature of 279° K. After matching the experimental data with an appropriately weighted combination of a 279° K blackbody curve and a laboratory reflection spectrum of lichens, he deduced that the thermal emission was a minor contribution, rising to perhaps 33 per cent of the total intensity at 3.8 μ . This result was adopted in the interpretation of his 1958 observations.

To obtain a feeling for the intensities involved we have plotted three curves in figure 10 - the blackbody curves for 255 and 300°K and the reflected intensity for a Martian surface element perpendicular to the sun, assuming a 5500°K sun. To calculate the latter we assumed an arbitrary reflectivity of 0.01, a value which may be somewhat low. For the emission curves an emissivity of 1 was adopted so that they are probably a few per cent high for the Martian surface. The spectra presented by Sinton, figure 1, are essentially effective reflectivities. However since the solar intensity varies only slightly over the wavenumber range covered they also are good representations of the energy leaving the surface.

Several points of interest now arise. It is seen that, even for a temperature of 255°K, the thermal emission can make a very significant contribution. We have not attempted to calculate effective temperatures for Sinton's two Martian spectra, figure 1, but we would estimate from Gifford's isotherms (9) and Sinton's 1956 calculated effective temperature of 279°K that 255°K is not excessively high. It appears then that the contribution of the reflected radiation, at least near the Mars I band, is comparable to the thermal emission.

This raises the question of the band intensities. It is expected that the emission spectrum will be relatively featureless. What features that are present will be opposite to the reflection features and will tend to smooth the Martian curve. It follows that the pure reflection spectrum would have spectral features more pronounced than any observed spectrum which is the resultant of both reflection and emission. In particular the relative intensity of the Mars I band in a pure reflection spectrum would be greater.

When in addition one considers the large bright areas included in the nominal Syrtis Major spectrum it is clear that the spectral features would be more intense if only the dark area were observed. At least this is true if the features are attributed to effects solely of the dark areas as Sinton's measurements indicate. This makes the assignment of the Mars I band, the one most affected by these considerations, still more difficult since we have observed in the laboratory no intense features in this region. It also stipulates that if organic matter is responsible for the other two, the material must have a very high surface concentration.

There is an alternative to the contribution of emission as an explanation for the intensity increase in the Martian reflection spectrum at lower wavenumbers. All of the samples we have examined which contained water show such an increase due to the wing of the very intense water band at 3400 cm⁻¹. Samples containing no water, such as paraffin wax, do not exhibit this effect. If the increase in the Mars spectra is not due to the characteristics of the emitted radiation it may arise from water in the surface material. The water could be in the liquid state in plants, in minerals as water of crystallization, or adsorbed on the surface.

We unfortunately have no answers for the major questions raised here. At the present we know of no satisfactory explanation of the Martian bands. In view of their potentially great significance with respect to a Martian biology further work is clearly indicated. In addition to further laboratory studies, observations of the planet with improved spectral and spatial resolution would be extremely valuable. In conjunction with radiometric temperature measurements they could possibly define the problem sufficiently to enable a solution to be found.

VI. Evidence for Life on Mars

In 1954, de Vaucouleurs (10) wrote, "As for the idea, so often expressed, of a different kind of life adapted to such conditions (i.e. on Mars) - what might be termed a 'generalized' life - it remains to a large extent beyond the realm of positive science."

This approach does not seem to have had much appeal, and most authors since then have stressed the positive aspects, generally overlooking contradictory observations. Indeed, the reader would undoubtedly conclude that various observations of Mars indicate considerably better than an even chance for the existence of life there. The opinion of the present

authors is that the evidence has been distorted and that a reexamination is required. A study of the scope necessary will not be undertaken here, rather we will restrict ourselves to a few observations intended to illustrate our point.

The observational arguments for the existence of Martian life are 1) the various colors, including green, exhibited by the dark areas, 2) the seasonal changes of the dark areas, 3) the ability of the dark areas to regenerate after an extensive "dust storm," and 4) the presence of 2700-3000 cm⁻¹ "absorption" bands, attributed to organic molecules.

We have already shown that the last, most recent point, is at least doubtful. The argument on the basis of the colors is also inconclusive. Kuiper (6) demonstrated the absence of the near infrared reflection maximum, characteristic of most green plants, indicating that chlorophyll was not responsible for the color. In this context Sinton has noted that this does not exclude chlorophyll since there may be present other pigments absorbing the near infrared radiation. This would actually be expected since any Martian plants would probably evolve into forms reflecting the barest minimum of the sparse solar radiation available. It has been suggested by many workers that the color is a contrast effect with the bright reddish continents. The meager quantitative data have been discussed by Opik (11) who has reduced photometric observations of Kozyrev on the very dark area Syrtis Major to intrinsic reflectivities by factoring out the estimated atmospheric attenuation and reflectivity. The result is that this area is bluer than a continent, but its color is still reddish. His reduction is questionable since he assumes the surface scattering properties to be similar to the moon in the sense that the disk would be uniformly bright if no atmosphere were present. However the qualitative conclusion that Syrtis Major has a red tint seems reasonable. While these considerations all weigh against a clorophyllbased plant life, the lichen systems suggested by Kuiper (but criticized by Salisbury) can satisfy the observed colors, real or imaginary. The broad leaved plants envisaged by Salisbury must have a chemical composition completely unlike any such terrestrial species, but this is to be expected. The significant point is that there is no positive colorimetric evidence pointing to forms of vegetation with which we are familiar.

The second and third arguments remain as the most cogent. To us the second loses some of its allure when one considers the severity of the

Martian climate. Focas (12) has photometered several dark regions on Mars from plates taken over a Martian year. All reveal the seasonal change with a distinct darkening wave moving from pole to pole with the seasons. An area showing one of the greatest seasonal changes is Depressio Hellespontica, located at about 60-65° latitude. Now the temperatures at this location, according to the data of Lampland as reduced by Gifford (9) reaches a maximum of -23°C for the southern summer. This temperature has an uncertainty due, among other factors, to the assumptions that the surface radiates as a blackbody and the atmosphere is transparent. The latter is reasonably valid for the 8-13 u terrestrial window used for the observations. but the first has been questioned by Opik who believes the emissivity to be closer to 0.75 and to 1, "by analogy with terrestrial substances." The net effect is to raise the Martian temperatures by about 5 per cent, i.e. ca. 13°C, making the environment more hospitable to life. Actually there are few literature data on the emissivity of terrestrial surfaces, but at least one report (13) indicates that the emissivities are higher. Even accepting this low emissivity and possible systematic errors it seems unlikely that the summer temperature of Depressio Hellespontica could be raised to 0°C. Accepting this, and knowing the uninviting composition of the atmosphere, we consider it highly improbable that any life, based on water as solvent, can exist in this region with the activity necessary to explain the darkening variations. Schemes can no doubt be envisaged to circumvent these objections but they must surely be very special. If a non-life mechanism must be invoked to explain the seasonal change of this one dark area, it is reasonable to assume it is applicable to the other dark regions and that this property cannot be used as an indication of life, even at the equator where the temperatures are more equable.

The rejuvenation property of the dark areas is one aspect which can be basically accepted as pertinent, although, as Salisbury has pointed out, a lichen cover would not be expected to behave in this manner. Higher forms of plant life such as Salisbury favors would possess this property.

While certain observations seem to favor life, others are very difficult to explain by this hypothesis. The normally bright region Hellas was dark during the 1954 opposition but returned in 1956 to its position as the second brightest area on the planet, only the polar caps exceeding it in reflectivity (14,15). Any form of plant life which, under Martian conditions, can so rapidly expand over and then retreat from an area of 290,000 square miles

must be truly unique. Other nonseasonal changes in the extent of the dark and bright areas are equally difficult to explain by a life hypothesis.

As an alternative to the life interpretation, we prefer an inorganic explanation based on ideas of McLaughlin (15) and Kuiper (16). Assuming volcanoes, both active and dead, and suitable winds the phenomena can be interpreted in a reasonably consistent manner. Thus the temporary darkening of Hellas would be ascribed to a film of volcanic ash produced by an active volcano just prior to the 1954 observations. This volcano then became passive and the winds proceeded to scour the area (McLaughlin considers it to be a high plateau since "it is often snow-covered, and in winter forms a vast extension of the polar cap down to latitude 30°"), restoring it to its previous brightness. Other non-periodic changes would be explained in like manner, invoking appropriate combinations of volcanic ash, lava and winds.

The regenerative feature according to Kuiper is then achieved by atmospheric circulation. Presumably this involves dust being blown on to the dark areas (postulated by Kuiper to be lava and not ash) during the late summer, fall, and winter, and then removed during the spring. Opik objects to such an interpretation, his principal argument being that radiometric and polarimetric measurements indicate that "the soil of the dark areas seems to be of the same grainy nature as that of the bright areas." This is not convincing since volcanic lava can have optical and thermal properties which are not dissimilar to those of the dark areas--similarity of certain optical and thermal properties for two materials does not guarantee that they are of the same nature. Opik also states that the presence of life is indicated by the permanence of the dark markings inspite of the numerous extensive dust storms. Without a regenerative property "during millions of centuries the dust storms would have covered the entire planet with a deep layer of dust...." The significance of this statement is decreased by his acceptance of a surface which has a non-uniform elevation (also see Dollfus (14)).

This is intended not as a complete treatment of the volcanic-wind model but as an indication of its real possibilities. We are inclined to favor it although not rejecting the life interpretation. But we would at least extend de Vaucouleurs' observation to 1963 and say again that discussion of life on Mars is still "... to a large extent beyond the realm of positive science."

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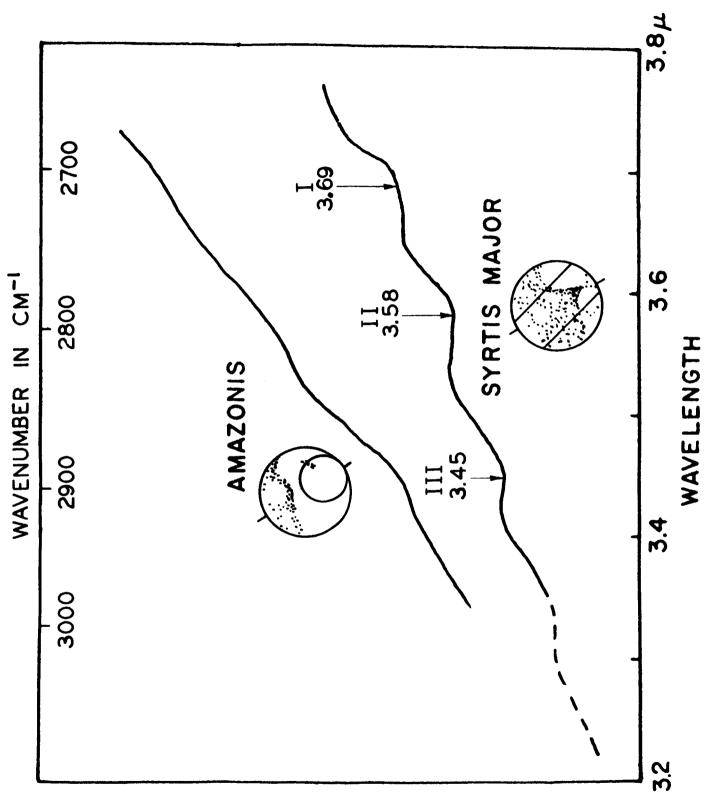


Figure 1. The reflection spectra of Mars as obtained by W. M. Sinton. The curves shown are the result of dividing the Martian spectra by the spectrum of the sun. The inserts indicate the size and location of the spectrum of the sun. aperture on the disk.

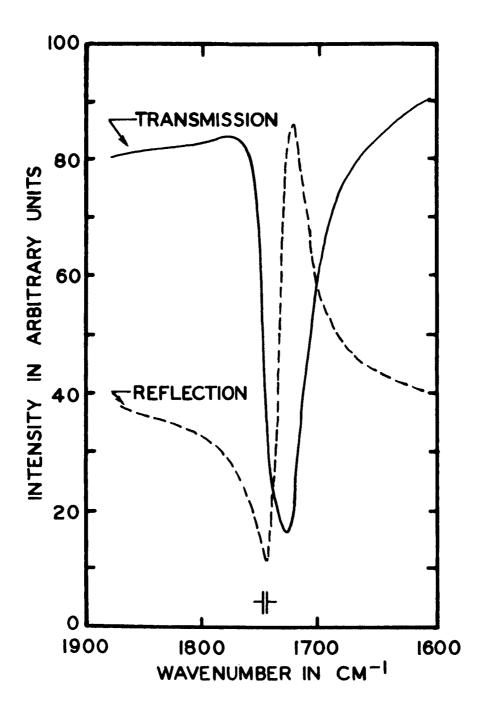
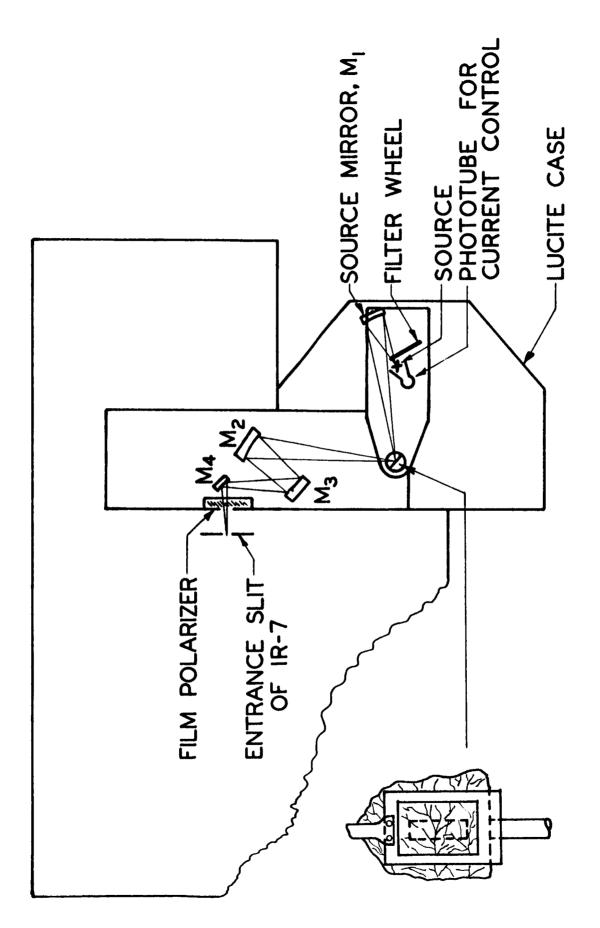


Figure 2. The transmission and reflection spectra of the C=O band of lucite. A film 25 μ thick was observed in transmission, a block 1 cm. thick in reflection.



The arm holding the source, filter Figure 3. The single beam accessory used to record reflection spectra for different angles of incidence and reflection. The sample can be rotated about its perpendicular axis. The arm holding the source, f wheel, etc., can be rotated 1800 about the same axis.

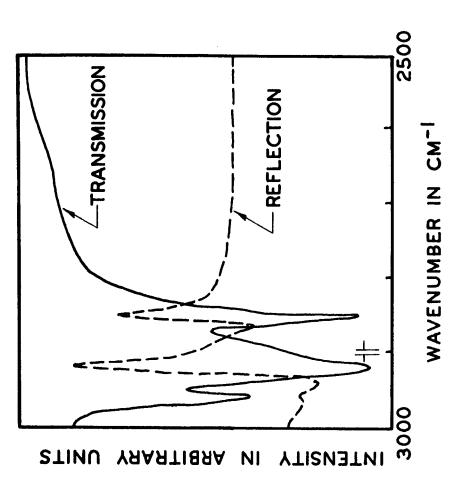


Figure 4. The transmission and reflection spectra of paraffin wax.

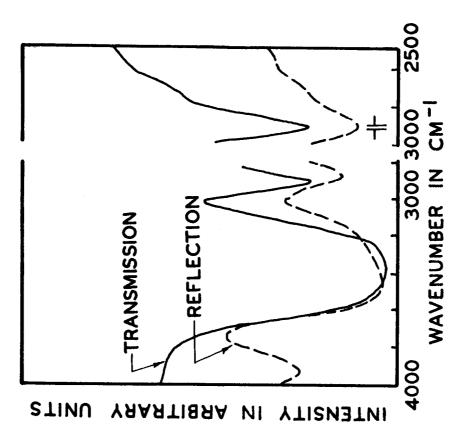


Figure 5. The transmission and reflection spectra of cellulose.

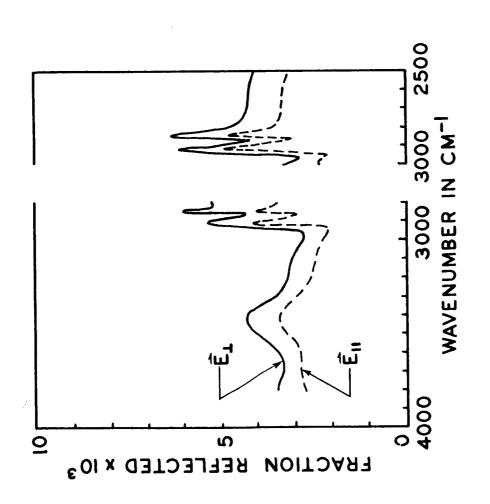


Figure 5. The reflection spectra of an Agapanthus leaf for the two polarizations perpendicular and parallel to the plane of incidence. The angle of incidence was 20°. The spectra were obtained using the single beam accessory.

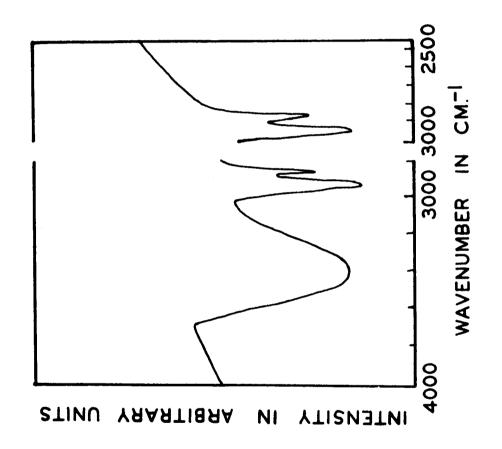


Figure 7. The transmission spectrum of the epidermis and cuticle of an Agapanthus leaf.

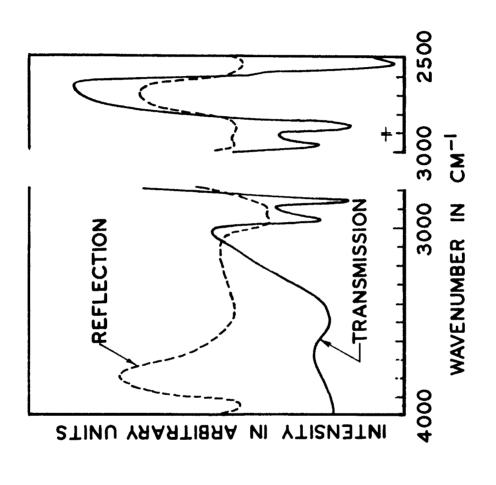
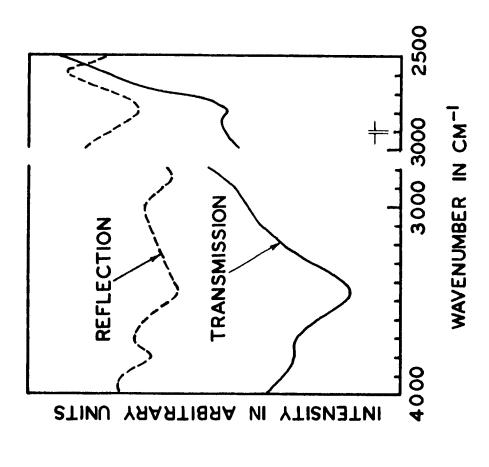


Figure 8. The transmission and reflection spectra of calcite, $CaCO_3$.



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Figure 9. The transmission and reflection spectra of $PbCO_3$.

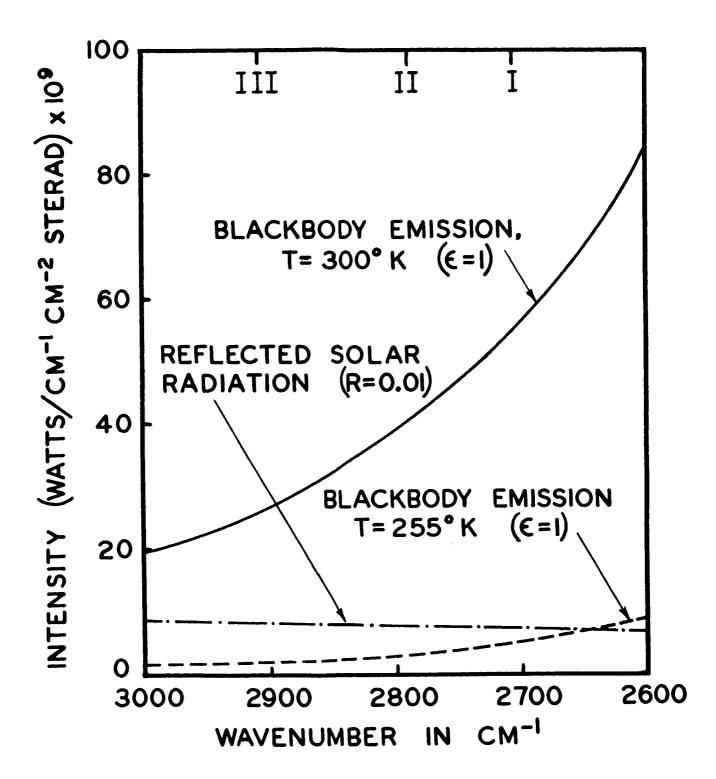


Figure 10. Calculated intensities of the reflected and emitted radiation for a Martian surface element perpendicular to the sun.